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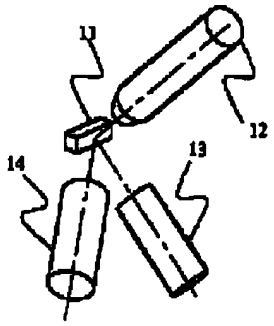
(54) METHOD AND APPARATUS FOR MEASUREMENT OF FILM THICKNESS

(57) Abstract:

PROBLEM TO BE SOLVED: To provide a method in which the film thickness of a sample whose film is being formed by a vacuum evaporation operation is measured and controlled with accuracy at one atomic layer thickness by an apparatus configuration which is low-cost and whose structure is comparatively simple and to provide its apparatus.

SOLUTION: An apparatus is composed of a substrate 11, of an electron gun 12 by which the substrate 11 is irradiated with primary electrons, of a vapor deposition device 13 by which a sample is vapor-deposited on the substrate 11 and of a secondary electron detector 14 which measures the intensity of secondary electrons sputtered by the primary electrons. The electron gun has a function to freely set the energy of the primary electrons from about 10eV up to about 5keV. At this time, the angle of incidence on the sample of the primary electrons is larger than an angle of total reflection.



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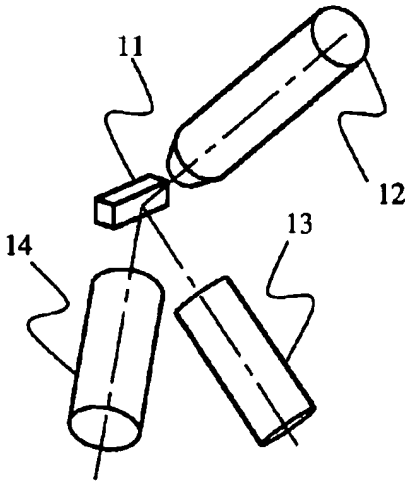
(54)【発明の名称】　膜厚計測法および装置

(57)【要約】

【目的】　安価で構造が比較的簡単な装置構成で、真空蒸着による成膜中の試料の膜厚を１原子層厚精度で測定および制御する方法、およびその装置を提供すること。

【構成】　基板11、およびこの基板11に１次電子を照射するための電子銃12、基板11上に試料を蒸着する蒸着装置13、１次電子によりたたき出された２次電子の強度を測定するための２次電子検出器14からなる。電子銃12は１次電子のエネルギーを10eV程度から5keV程度まで自由に設定できる機能を持つ。このとき、１次電子の試料への入射角は全反射角よりも大きい。

図１



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【特許請求の範囲】

【請求項１】真空蒸着による薄膜試料作製中に、試料表面へ１次電子を照射することにより放出される２次電子の強度が、試料の膜厚の関数として１原子層厚の周期で振動する現象を利用した膜厚測定法において、１次電子の試料表面から測った入射角を、電子の試料に対する全反射角よりも大きくすることを特徴とする膜厚計測法。

【請求項２】２次電子の強度振動が得られる条件において現れる試料への吸収電流振動を利用する請求項１記載の膜厚計測法。

【請求項３】強度振動測定を行う２次電子のエネルギーをエネルギー分析器によって選別することにより、振動が最も明瞭にあらわれるエネルギーを持った２次電子だけを用いる請求項１記載の膜厚計測法。

【請求項４】１次電子エネルギーを任意の値に設定して２次電子強度振動もしくは吸収電流振動が最も明瞭に現れる１次電子のエネルギーを選別して利用する請求項１もしくは請求項２記載の膜厚計測法。

【請求項５】真空蒸着による薄膜試料が表面に形成されるための基板、前記試料表面へ１次電子を照射する電子銃、前記１次電子の照射により試料表面より放出される２次電子を検出する２次電子検出器を備え、前記電子銃の試料表面への１次電子の照射角が試料表面から見て全反射角より大きい角度となるように設定されていることを特徴とする膜厚計測装置。

【請求項６】前記２次電子に代えて、試料への吸収電流振動を検出する請求項５記載の膜厚計測装置。

【請求項７】前記検出２次電子のエネルギーを分析するためのエネルギー分析器を備える請求項５記載の膜厚計測装置。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は、分子線エピタキシーなどの真空蒸着法による成膜中に、試料の膜厚を１原子層厚以下の精度で計測する方法および装置に関する。

【０００２】

【従来の技術】真空蒸着法により試料を成膜する際、２次電子の強度が１原子層厚の周期で振動する現象を利用することが可能だと考えられるが、この現象は、従来、反射高速電子回折の実験条件で観測されており、ジャーナル・オブ・クリスタル・グロウス（Journal of Crystal Growth）81巻（1987年）55頁から58頁に報告されている。

【０００３】

【発明が解決しようとする課題】しかし、上記方法は、１次電子を試料表面すれすれ（試料表面から測って、電子の全反射角程度以下。以下、角度はすべて試料表面から測るものとする）に入射させることを必要とするため、電子の試料への入射を妨げないような特殊な試料台、および１次電子の入射角を調整するための機構が試

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料台もしくは電子銃に必要となるため、装置全体が大型化および複雑化し、これによって装置の取扱が困難になる欠点および装置全体の価格が高価になる欠点があった。

【０００４】発明が解決しようとする課題は、安価で構造が単純かつコンパクトな装置を用いて、真空蒸着による成膜中の試料の膜厚を１原子層厚以下の精度で測定および制御することにある。

【０００５】

【課題を解決するための手段】上記の問題点は、１次電子の試料への入射角を全反射角よりも大きな角度とすることで解決できる。

【０００６】

【作用】従来の技術において、反射高速電子回折の実験条件で観測されている２次電子の強度振動は、サーフェイス・サイエンス・レターズ（Surface Science Letters）245巻（1991年）L159頁からL162頁に解説されている内容を用いて、次のように説明できる。

【０００７】真空蒸着中の試料が層状成長している場合、その表面に形成されるステップの密度は１原子層周期で振動する。１次電子を試料に全反射角以内の角度で入射させても、試料表面にステップが存在する場合、その端から電子は試料内に侵入できる。侵入した電子の一部は試料内部での回折によって表面へ向うが、さらにその一部が試料表面での全反射によって再び試料内部に向い吸収される。よってステップ密度が振動すれば試料に吸収される電子数も振動することになる。２次電子数は１次電子数と試料に吸収される電子数の差であるから、試料に吸収される電子数が変化すれば、２次電子強度も変化することになる。

【０００８】以上により従来の技術では、１次電子の試料への入射角が全反射角よりも大きい場合、２次電子振動は観測されない、と考えられてきた。

【０００９】しかし、発明者の研究結果によれば、次の原理により、１次電子の試料への入射角が全反射角よりも大きい場合でも、２次電子振動が観測され得る。すなわち、２次電子の収率は試料表面の仕事関数が小さいと大きくなるから、仕事関数が振動すれば２次電子強度も振動することになる。蒸着中の試料表面の仕事関数の振動は、サーフェイス・サイエンス（Surface Science）298巻（1993年）173頁から186頁に解説されている。

【００１０】また、同じ現象を次のように説明することもできる。すなわち、基板上に試料を蒸着する場合、試料が薄膜であるとその中に量子井戸が形成される。この量子井戸状態は試料と基板の界面および試料表面の２つの境界条件で規定される。ここで前者の境界条件は試料膜厚に依存しないと考えられるが、後者の境界条件である表面形状（荒さ）はステップ密度に応じて１原子層周期で変化し、薄膜試料内の電子の状態密度、したがってこれを反映する２次電子強度もこの周期で変化する。

【００１１】発明者のこの研究結果によれば、１次電子の試料への入射角が全反射角よりも大きい場合でも、基板への試料の蒸着中に、２次電子振動が試料の層成長の周期と同期して振動するので、これにより蒸着中の試料の膜厚を１原子層厚以下の精度で知ることが可能となる。ここで、１次電子のエネルギーは、２次電子励起が可能となる最低のエネルギーである10eV程度よりも大きければ、特に制約はない。

【００１２】

【実施例】図１は本発明による膜厚測定装置の実施例の第一の基本構成を示す図である。本構成は基板11、およびこの基板11に１次電子を照射するための電子銃12、基板11上に試料を蒸着する蒸着装置13、１次電子によりたたき出された２次電子の強度を測定するための２次電子検出器14からなる。ここで電子銃12は１次電子のエネルギーを10eV程度から5keV程度まで自由に設定できる機能を持つものとする。また、１次電子の試料への入射角は全反射角よりも大きければ、特に制約はない。

【００１３】この構成では、基板11上に蒸着装置13を用いて試料を蒸着しながら、電子銃12によって約1keVのエネルギーを有する１次電子を照射する。このとき基板11上に形成された薄膜試料表面から放出される２次電子の強度を２次電子検出器14により測定する。測定された２次電子強度は基板11上に形成される試料の膜厚の関数として１原子層の周期で振動する。試料として金を蒸着したときの２次電子強度の振動を図２に示す。この振動の周期Ｔは試料の１原子層の厚さに等しいため、横軸である金の厚さをＴで校正でき、正確な金薄膜の厚さがわかる。従って、この現象を用いることで高精度の膜厚計測が可能となる。

【００１４】さらに、２次電子強度振動が最も顕著に現れる１次電子のエネルギーは試料の種類や面方位などによって異なる場合がある。本実施例では例として１次電子のエネルギーを1keV程度としたが、試料の種類や面方位に応じて１次電子のエネルギーを変えることで、２次電子強度振動が最も顕著に現れる最適の条件で本方法を用いればよい。

【００１５】図３は本発明による膜厚測定装置の実施例の第二の基本構成を示す図である。本構成は基板11、およびこの基板11に１次電子を照射するための電子銃12、基板11上に試料を蒸着する蒸着装置13、１次電子によりたたき出された２次電子を収集し効率よく伝送するための電子レンズ31、２次電子のエネルギーを選別するためのエネルギー分析器32、エネルギー分析された後の２次電子強度を測定するための２次電子検出器14からなる。さらに電子銃12は第一の実施例と同様に、１次電子のエネルギーを10eV程度から5keV程度まで自由に設定できる機能を持つものとする。また、第一の実施例と同様に、１次電子の試料への入射角は全反射角よりも大きければ、特に制約はない。

【００１６】この構成では、基板11上に蒸着装置13により試料を蒸着しながら、電子銃12によって約1keVのエネルギーを有する１次電子を照射する。このとき基板11上に形成された薄膜試料表面から放出される２次電子を電子レンズ31によって収集し、エネルギー分析器32へ効率よく伝送する。エネルギー分析器32に入射した２次電子のうち特定のエネルギーを持った電子だけが２次電子検出器14に導かれ、その強度が測定される。測定された２次電子強度は第一の実施例と同様に、基板11上に形成される試料の膜厚の関数として振動し、その振動の周期Ｔが試料の１原子層の厚さに等しいため、この振動をモニタすることで高精度の膜厚計測が可能となる。

【００１７】さらに、２次電子強度振動が最も顕著に現れる２次電子のエネルギー範囲は試料の種類や面方位などによって異なる場合がある。本実施例ではエネルギー分析器32によって特定のエネルギーを持った２次電子だけを２次電子検出器14に導くことができるので、これにより２次電子強度振動が最も顕著に現れるエネルギーを選び、その２次電子の強度を測定すればよい。

【００１８】さらに、第一の実施例と同様に、２次電子強度振動が最も顕著に現れる１次電子のエネルギーは試料の種類や面方位などによって異なる場合があるため、本実施例でも試料の種類や面方位に応じて１次電子のエネルギーを調整することで、２次電子強度振動が最も顕著に現れる最適の条件で本方法を用いればよい。

【００１９】図４は本発明による膜厚測定装置の実施例の第三の基本構成を示す図である。本構成は基板11、およびこの基板11に１次電子を照射するための電子銃12、基板11上に試料を蒸着する蒸着装置13からなる。さらに電子銃12は第一の実施例と同様に、１次電子のエネルギーを10eV程度から5keV程度まで自由に設定できる機能を持つものとする。また、第一の実施例と同様に、１次電子の試料への入射角は全反射角よりも大きければ、特に制約はない。

【００２０】この構成では、基板11上に蒸着装置13を用いて試料を蒸着しながら、電子銃12によって約1keVのエネルギーを有する１次電子を照射する。このとき基板11において測定される吸収電流は基板11上に形成される試料の膜厚の関数として１原子層の周期で振動する。この振動の様子は図２に示した２次電子強度の振動と同様である。この振動の周期Ｔは２次電子強度の振動の周期と同じく試料の１原子層の厚さに等しいため、横軸である金の厚さをＴで校正でき、正確な金薄膜の厚さがわかる。従って、この現象を用いることでも高精度の膜厚計測が可能となる。本実施例では第一の実施例とは異なり、２次電子検出器を必要としないので、より簡便な膜厚計測装置の実現が可能となる。

【００２１】さらに、吸収電流振動が最も顕著に現れる１次電子のエネルギーは２次電子強度振動と同様に、試料の種類や面方位などによって異なる場合がある。本実

施例では例として1次電子のエネルギーを1keV程度としたが、試料の種類や面方位に応じて1次電子のエネルギーを変えることで、吸収電流振動が最も顕著に現れる最適の条件で本方法を用いればよい。

【0022】図5は本発明による膜厚測定装置の実施例の第四の基本構成を示す図である。本構成は基板11、およびこの基板11に1次電子を照射するための電子銃12、基板11上に試料を蒸着する蒸着装置13、1次電子によりたたき出された2次電子の強度を測定するための2次電子検出器14、およびこれら装置の制御とデータ収集を行うためのコンピュータ51からなる。さらに電子銃12は第一の実施例と同様に、1次電子のエネルギーを10eV程度から5keV程度まで自由に設定できる機能を持つものとする。また、第一の実施例と同様に、1次電子の試料への入射角は全反射角よりも大きければ、特に制約はない。

【0023】この構成では、基板11上に蒸着装置13により試料を蒸着しながら、電子銃12によって約1keVのエネルギーを有する1次電子を照射する。このとき基板11上に形成された薄膜試料表面から放出される2次電子の強度を2次電子検出器13により測定する。測定された2次電子強度は第一の実施例と同様に、基板11上に形成される試料の膜厚の関数として振動し、その振動の周期Tが試料の1原子層の厚さに等しいため、これにより高精度の膜厚計測が可能となる。この2次電子強度振動の信号をコンピュータ51によりモニタすることで、膜厚測定が自動化される。

【0024】さらに、本実施例では第一の実施例と同様に、2次電子強度振動が最も顕著に現れる1次電子のエネルギーは試料の種類や面方位などによって異なる場合があるため、本実施例でも試料の種類や面方位に応じて1次電子のエネルギーを調整することで、2次電子強度振動が最も顕著に現れる最適の条件で本方法を用いればよい。このとき2次電子強度振動測定および電子銃12の制御をコンピュータ51で行うことにより自動化が可能となる。

【0025】さらに、本実施例では蒸着時間に対する膜厚をコンピュータ51でモニタし、その蒸着速度が任意の速度になるように蒸着装置13を制御することにより、蒸着速度制御を自動化できる。

【0026】さらに、本実施例では蒸着時間に対する膜厚をコンピュータ51でモニタし、目的とする膜厚が得られたところで蒸着をコンピュータ51により自動停止することで、任意の膜厚の試料作製が自動的にできる。このとき目的とする膜厚の情報はあらかじめコンピュータ51に入力しておけばよい。

【0027】さらに、2次電子強度振動が最も顕著に現れる2次電子のエネルギー範囲は試料の種類や面方位などによって異なる場合がある。従って本実施例でも第二の実施例と同様に、エネルギー分析器を利用することで特定のエネルギーを持った2次電子だけを2次電子検出器14に導き、その2次電子の強度を測定すればよい。このエネルギー選別もコンピュータ51により自動化することができる。

【0028】さらに、本実施例では2次電子強度振動を利用したが、第四の実施例に示した吸収電流振動を利用することもできる。この場合、2次電子検出器14は不要となり、装置全体の構成はさらに簡便なものとなる。

【0029】なお、上述の説明では、試料に対する電子線の照射は、試料の全面に対して行なうこととしたが、電子線を絞って、試料の特定の領域での膜厚測定とする場合でも同様に実施できる。

【0030】

【発明の効果】本発明によれば、1次電子の入射角を全反射角よりも大きくして試料に照射することで、真空蒸着による成膜中の試料の膜厚を1原子層厚以下の精度で測定および制御できるので、装置全体の構成が簡略化され、取り扱いが容易になり装置全体の価格も安価となる効果がある。さらに本発明によれば、反射高速電子回折を用いた従来装置よりも構造が比較的簡単な低エネルギー用の電子銃を用いているため、専用の高電圧電源や電子銃、電子光学系を用いるのに比べ装置全体が安価なシステムとなる効果がある。これらの効果の学術分野への応用、さらにはその工業的価値は非常に高いものである。

【図面の簡単な説明】

【図1】本発明の基本構成を表す図。

【図2】本発明により得られた2次電子強度振動の図。

【図3】本発明とエネルギー分析器の組み合わせを示す図。

【図4】本発明に吸収電流振動を利用した場合の構成を表す図。

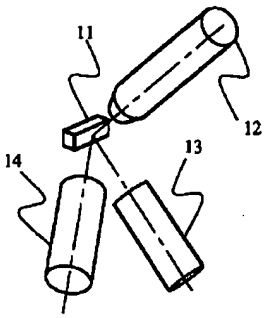
【図5】本発明とコンピュータによる蒸着の自動制御を示す図。

【符号の説明】

11･･･基板、12･･･電子銃、13･･･蒸着装置、14･･･2次電子検出器、31･･･電子レンズ、32･･･エネルギー分析器、51･･･コンピュータ。

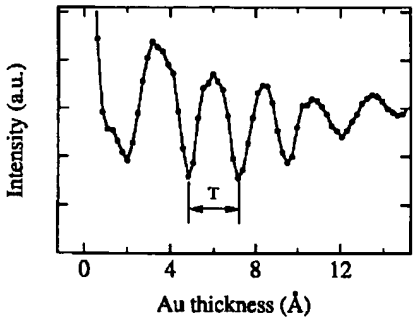
【図1】

図1



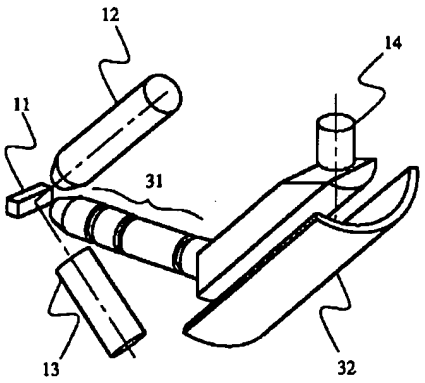
【図2】

図2



【図3】

図3

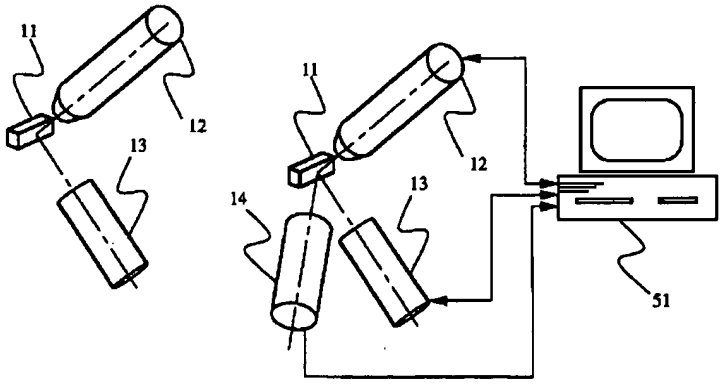


【図4】

図4

【図5】

図5



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(54) [Title of the Invention] METHOD AND APPARATUS FOR MEASUREMENT OF FILM THICKNESS

(57) [Abstract]

[Object] [The object of the present invention is] to provide a method and apparatus for measuring and controlling, with monatomic layer thickness precision, the film thickness of a sample during film formation by vacuum deposition, by means of an apparatus construction which is inexpensive and which has a relatively simple structure. [Constitution] [The apparatus] comprises a substrate 11, an electron gun 12 which is used to irradiate this substrate 11 with primary electrons, a vapor deposition apparatus 13 which deposits a sample on this substrate 11 by vapor deposition, and a secondary electron detector 14 which is used to measure the intensity of the secondary electrons that are knocked out by the primary electrons. The electron gun 12 has a function that allows the energy of the primary electrons to be freely set from approximately 10 eV to approximately 5 keV. In this case, the angle of incidence of the primary electrons on the sample is greater than the total-reflection angle.

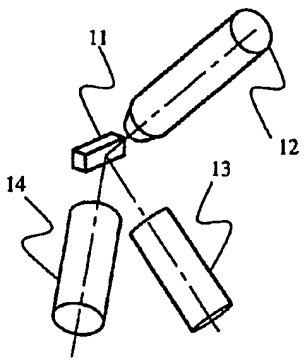

Figure 1

[Claims]

[Claim 1] A film thickness measurement method which is characterized by the fact that in a film thickness measurement method which utilizes the phenomenon whereby the intensity of the secondary electrons that are emitted as a result of the irradiation of the sample surface with primary electrons during the manufacture of a thin film sample by vacuum deposition oscillates with the period of the monatomic layer thickness as a function of the film thickness of the sample, the angle of incidence of the primary electrons as measured from the sample surface is set at an angle that is greater than the total-reflection angle of the electrons with respect to the sample.

[Claim 2] The film thickness measurement method according to Claim 1, wherein the oscillation of the absorption current into the sample that appears at the conditions under which the [above-mentioned] intensity oscillation of the secondary electrons is obtained is utilized.

[Claim 3] The film thickness measurement method according to Claim 1, wherein only secondary electrons having an energy at which the [above-mentioned] oscillation appears most clearly are used by selecting, by means of an energy analyzer, the energy of the secondary electrons for which the [above-mentioned] intensity oscillation measurement is performed.

[Claim 4] The film thickness measurement method according to Claim 2, wherein the energy of the primary electrons is set at an arbitrary value, and the energy of the primary electrons at which the secondary electron intensity oscillation or absorption current oscillation appears most clearly is selected and utilized.

[Claim 5] A film thickness measurement apparatus which is characterized by the fact that the apparatus comprises a substrate which is used for the formation of a thin film sample (on the surface of this substrate) by means of vacuum deposition, an electron gun which irradiates the surface of the above-mentioned sample with primary electrons, and a secondary electron detector which detects the secondary electrons that are emitted from the surface of the sample as a result of the above-mentioned irradiation with primary electrons, and the angle of irradiation of the surface of the sample with the primary electrons by means of the above-mentioned electron gun is set so that this angle is larger than the total-reflection angle as seen from the surface of the sample.

[Claim 6] The film thickness measurement apparatus according to Claim 5, wherein the oscillation of the absorption current into the sample is detected instead of the above-mentioned secondary electrons.

[Claim 7] The film thickness measurement apparatus according to Claim 5, which comprises an energy analyzer that is used to analyze the energy of the above-mentioned detected secondary electrons.

[Detailed Description of the Invention]

[0001]

[Field of Industrial Utilization] The present invention relates to a method and apparatus for measuring the film thickness of the sample during film formation by a vacuum deposition method such as molecular beam epitaxy with a precision corresponding to the thickness of a monatomic layer or better.

[0002]

[Prior Art] When a sample film is formed by a vacuum deposition method, it is conceivable that it might be possible to utilize the phenomenon whereby the intensity of the secondary electrons oscillates with the period of a monatomic layer thickness. This phenomenon has conventionally been observed under experimental conditions in reflective high-speed electron diffraction, and has been reported in *Journal of Crystal Growth*, Vol. 81 (1987), pp. 55-58.

[0003]

[Problems that the Invention is to Solve] In the above-mentioned method, however, it is necessary to cause the primary electrons to be incident at an angle that just grazes the sample surface (i.e., an angle that is approximately equal to or less than the total-reflection angle of the electrons as measured from the sample surface; below, all angles are assumed to be angles measured from the sample surface). Consequently, a special sample holder which does not interfere with the incidence of the electrons on the sample, and a mechanism for adjusting the angle of incidence of the primary electrons, are required in the sample holder or electron gun, so that the overall apparatus is increased in size and complexity. As a result, such a method suffers from the drawback of difficult handling of the apparatus and high cost of the apparatus as a whole.

[0004] The object of the present invention is to measure and control the film thickness of the sample during film formation by vacuum deposition with a precision corresponding to the thickness of a monatomic layer or better using an inexpensive apparatus which has a simple and compact structure.

[0005]

[Means for Solving the Problems] The above-mentioned problems are solved by setting the angle of incidence of the primary electrons on the sample at an angle that is greater than the total-reflection angle.

[0006]

[Operation] The intensity oscillation of the secondary electrons observed under experimental conditions in reflective high-speed electron diffraction in the prior art may be described as follows using the content of *Surface Science Letters*, Vol. 245 (1991), pp. L159-L162.

[0007] In cases where a sample in vacuum deposition is grown in layer form, the density of the step formed on the sample surface oscillates with the period of a monatomic layer. In cases where a step is present on the sample surface, the electrons can invade the interior of the sample via the end [of this step] even if the primary electrons are caused to be incident on the sample at an angle that is equal to or less than the total-reflection angle. Some of the invading electrons are directed toward the surface by diffraction in the interior portions of the sample; furthermore, some of these electrons are again directed toward the interior of the sample by total reflection at the sample surface, and are absorbed. Accordingly, if the step density oscillates, the number of electrons absorbed by the sample also oscillates. Since the number of secondary electrons is the difference between the number of primary electrons and the number of electrons absorbed by the sample, if the number of electrons absorbed by the sample varies, the secondary electron intensity also varies.

[0008] Accordingly, in conventional techniques, it has been believed that no oscillation of secondary electrons is observed in cases where the angle of incidence of the primary electrons on the sample is greater than the total-reflection angle.

[0009] However, according to the results of research conducted by the inventors, an oscillation of secondary electrons can be observed on the basis of the following principle even in cases where the angle of incidence of the primary electrons on the sample is greater than the total-reflection angle. Specifically, since the yield of secondary electrons increases with a decrease in the work function of the sample surface, the secondary electron intensity also oscillates if there is an oscillation in the work function. The oscillation of the work function of the sample surface during vapor deposition is explained in *Surface Science*, Vol. 298 (1993), pp. 173-186.

[0010] Furthermore, the same phenomenon may be explained as follows. Specifically, in cases where a sample is formed on a substrate by vapor deposition, quantum wells are formed in this sample if the sample is a thin film. This quantum well state is regulated by two sets of boundary conditions, i.e., the boundary conditions of the interface between the sample and the substrate

and the boundary conditions of the surface of the sample. Here, it would appear that the former boundary conditions do not depend on the film thickness of the sample; however, the surface shape (roughness) constituting the latter boundary conditions varies with the period of a monatomic layer in accordance with the step density, and the state density of the electrons within the thin film sample, and therefore the secondary electron intensity reflecting this state density, also varies with this period.

[0011] According to the results of research on this subject conducted by the inventors, the secondary electron oscillation during the vapor deposition of the sample on the substrate oscillates in synchronization with the period of the layer growth of the sample even in cases where the angle of incidence of the primary electrons on the sample is greater than the total-reflection angle; as a result, the film thickness of the sample during vapor deposition can be ascertained with a precision corresponding to the thickness of a monatomic layer or better. Here, there are no particular restrictions on the energy of the primary electrons, as long as this energy is greater than approximately 10 eV, which is the lowest energy at which the excitation of secondary electrons is possible.

[0012]

[Embodiments] Figure 1 is a diagram which shows a first basic construction of an embodiment of the film thickness measurement apparatus of the present invention. This construction comprises a substrate 11, an electron gun 12 which is used to irradiate this substrate 11 with primary electrons, a vapor deposition apparatus 13 which forms a sample on the surface of the substrate 11 by vapor deposition, and a secondary electron detector 14 which is used to measure the intensity of the secondary electrons that are knocked out [of the sample] by the primary electrons. Here, the electron gun 12 has a function that allows the energy of the primary electrons to be freely set at values ranging from approximately 10 eV to approximately 5 keV. Furthermore, there are no particular restrictions on the angle of incidence of the primary electrons on the sample, as long as this angle of incidence is greater than the total-reflection angle.

[0013] In this construction, irradiation with primary electrons having an energy of approximately 1 keV is performed by means of the electron gun 12 while a sample is formed on the surface of the substrate 11 by vapor deposition using the vapor deposition apparatus 13. In this case, the intensity of the secondary electrons that are emitted from the surface of the thin film sample formed on the substrate 11 is measured by the secondary electron detector 14. The measured intensity of the secondary electrons oscillates with the period of a monatomic layer as a function of the film thickness of the sample that is formed on the substrate 11. Figure 2 shows

the oscillation of the secondary electron intensity in a case where gold is deposited as the sample by vapor deposition. Since the period T of this oscillation is equal to the thickness of a monatomic layer of the sample, the thickness of the gold shown on the horizontal axis can be calibrated by T, so that the accurate thickness of the gold thin film can be ascertained. Accordingly, high-precision film thickness measurements can be made by using this phenomenon.

[0014] Furthermore, there may be cases in which the energy of the primary electrons at which the oscillation of the intensity of the secondary electrons appears most conspicuously varies according to the type and surface orientation, etc., of the sample. In the present embodiment, the energy of the primary electrons was set at approximately 1 keV as an example; however, the present method may be used under optimal conditions at which the oscillation of the intensity of the secondary electrons appears most conspicuously by varying the energy of the primary electrons in accordance with the type and surface orientation of the sample.

[0015] Figure 3 is a diagram which shows a second basic construction of an embodiment of the film thickness measurement apparatus of the present invention. This construction comprises a substrate 11, an electron gun 12 which is used to irradiate this substrate 11 with primary electrons, a vapor deposition apparatus 13 which deposits a sample on the surface of the substrate 11 by vapor deposition, an electron lens 31 which is used to focus the secondary electrons that are knocked out [of the sample] by the primary electrons and to transmit these secondary electrons in an efficient manner, an energy analyzer 32 which is used to discriminate the energy of the secondary electrons, and a secondary electron detector 14 which is used to measure the intensity of the secondary electrons following the energy analysis. Furthermore, as in the first embodiment, the electron gun 12 has a function that allows the energy of the primary electrons to be freely set at an energy ranging from approximately 10 eV to approximately 5 keV. Moreover, as in the first embodiment, there are no particular restrictions on the angle of incidence of the primary electrons on the sample, as long as this angle of incidence is greater than the total-reflection angle.

[0016] In this construction, irradiation with primary electrons having an energy of approximately 1 keV is performed by means of the electron gun 12 while a sample is deposited on the surface of the substrate 11 by vapor deposition by means of the vapor deposition apparatus 13. In this case, the secondary electrons that are emitted from the surface of the thin film sample formed on the substrate 11 are focused by the electron lens 31, and are efficiently transmitted to the energy analyzer 32. Only those electrons having a specified energy (among the secondary electrons that are incident on the energy analyzer 32) are conducted to the

secondary electron detector 14, and the intensity of these secondary electrons is measured. As in the first embodiment, the measured intensity of the secondary electrons oscillates as a function of the film thickness of the sample that is formed on the surface of the substrate 11, and since the period T of this oscillation is equal to the thickness of a monatomic layer of the sample, high-precision film thickness measurements can be made by monitoring this oscillation.

[0017] Furthermore, there may be cases in which the secondary electron energy range in which the oscillation of the secondary electron intensity appears most conspicuously varies according to the type and surface orientation, etc., of the sample. In the present embodiment, since it is possible to conduct only secondary electrons that have a specified energy to the secondary electron detector 14 by means of the energy analyzer 32, it is sufficient to select in this way the energy at which the oscillation of the secondary electron intensity appears most conspicuously, and to measure this secondary electron intensity.

[0018] Furthermore, as in the first embodiment, there may be cases in which the energy of the primary electrons at which the oscillation of the intensity of the secondary electrons appears most conspicuously varies according to the type and surface orientation, etc., of the sample. Accordingly, in this embodiment as well, the present method may be used under optimal conditions at which the oscillation of the intensity of the secondary electrons appears most conspicuously by adjusting the energy of the primary electrons in accordance with the type and surface orientation of the sample.

[0019] Figure 4 is a diagram which shows a third basic construction of an embodiment of the film thickness measurement apparatus of the present invention. This construction comprises a substrate 11, an electron gun 12 which is used to irradiate this substrate 11 with primary electrons, and a vapor deposition apparatus 13 which deposits a sample on the surface of the substrate 11 by vapor deposition. Furthermore, as in the first embodiment, the electron gun 12 has a function that allows the energy of the primary electrons to be freely set at an energy ranging from approximately 10 eV to approximately 5 keV. Moreover, as in the first embodiment, there are no particular restrictions on the angle of incidence of the primary electrons on the sample, as long as this angle of incidence is greater than the total-reflection angle.

[0020] In this construction, irradiation with primary electrons having an energy of approximately 1 keV is performed by means of the electron gun 12 while a sample is deposited on the surface of the substrate 11 by vapor deposition using the vapor deposition apparatus 13. In this case, the absorption current that is measured in the substrate 11 oscillates with the period of a monatomic layer as a function of the film thickness of the sample that is formed on the

surface of the substrate 11. The conditions of this oscillation are the same as the conditions of the oscillation of the secondary electron intensity shown in Figure 2. Like the period of the oscillation of the secondary electron intensity, the period T of this oscillation is equal to the thickness of a monatomic layer of the sample; accordingly, the thickness of the gold shown on the horizontal axis can be calibrated by T, so that the accurate thickness of the gold thin film can be ascertained. Accordingly, high-precision film thickness measurements can be made by using this phenomenon. In this embodiment, unlike the first embodiment, no secondary electron detector is required; accordingly, a simpler film thickness measurement apparatus can be realized.

[0021] Furthermore, as in the case of the oscillation of the secondary electron intensity, there may be instances in which the energy of the primary electrons at which the oscillation of the absorption current appears most conspicuously varies according to the type and surface orientation, etc., of the sample. In the present embodiment, the energy of the primary electrons was set at approximately 1 keV as an example; however, the present method may be used under optimal conditions at which the oscillation of the absorption current appears most conspicuously by varying the energy of the primary electrons in accordance with the type and surface orientation of the sample.

[0022] Figure 5 is a diagram which shows a fourth basic construction of an embodiment of the film thickness measurement apparatus of the present invention. This construction comprises a substrate 11, an electron gun 12 which is used to irradiate this substrate 11 with primary electrons, a vapor deposition apparatus 13 which deposits a sample on the surface of the substrate 11 by vapor deposition, a secondary electron detector 14 which is used to measure the intensity of the secondary electrons that are knocked out [of the sample] by the primary electrons, and a computer 51 which is used to control these devices and to collect data. Furthermore, as in the first embodiment, the electron gun 12 has a function that allows the energy of the primary electrons to be freely set at an energy ranging from approximately 10 eV to approximately 5 keV. Moreover, as in the first embodiment, there are no particular restrictions on the angle of incidence of the primary electrons on the sample, as long as this angle of incidence is greater than the total-reflection angle.

[0023] In this construction, irradiation with primary electrons having an energy of approximately 1 keV is performed by means of the electron gun 12 while a sample is deposited on the surface of the substrate 11 by vapor deposition by means of the vapor deposition apparatus 13. In this case, the intensity of the secondary electrons that are emitted from the surface of the thin film sample that is formed on the surface of the substrate 11 is measured by

the secondary electron detector 13 [*sic*]*. As in the first embodiment, the measured intensity of the secondary electrons oscillates as a function of the film thickness of the sample that is formed on the surface of the substrate 11, and since the period T of this oscillation is equal to the thickness of a monatomic layer of the sample, high-precision film thickness measurements can be performed using this oscillation. Film thickness measurement can be automated by monitoring the signal of this secondary electron intensity oscillation by means of the computer 51.

[0024] Furthermore, in this embodiment, as in the first embodiment, there may be cases in which the energy of the primary electrons at which the oscillation of the secondary electron intensity appears most conspicuously varies according to the type and surface orientation, etc., of the sample. Accordingly, in this embodiment as well, the present method may be used under optimal conditions at which the oscillation of the intensity of the secondary electrons appears most conspicuously by adjusting the energy of the primary electrons in accordance with the type and surface orientation of the sample. In this case, automation can be accomplished by performing the measurement of the oscillation of the intensity of the secondary electrons and the control of the electron gun 12 by means of the computer 51.

[0025] Furthermore, in the present embodiment, control of the vapor deposition rate can be automated by monitoring the film thickness with respect to the vapor deposition time by means of the computer 51, and controlling the vapor deposition apparatus 13 so that the vapor deposition rate is maintained at an arbitrary rate.

[0026] Furthermore, in the present embodiment, the manufacture of samples with an arbitrary film thickness can be automatically performed by monitoring the film thickness with respect to the vapor deposition time by means of the computer 51, and automatically stopping vapor deposition using the computer 51 when the desired film thickness is obtained. In this case, information regarding the desired film thickness may be input into the computer 51 beforehand.

[0027] Furthermore, there may be cases in which the secondary electron energy range in which the oscillation of the intensity of the secondary electrons appears most conspicuously varies according to the type and surface orientation, etc., of the sample. Accordingly, in this embodiment as well, as in the second embodiment, it is possible to utilize the energy analyzer so that only secondary electrons that have a specified energy are conducted to the secondary electron detector 14, and so that the intensity of these secondary electrons is measured. This energy discrimination can also be automated by means of the computer 51.

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* Translator's note: apparent error in the original for "secondary electron detector 14."

[0028] Furthermore, the oscillation of the intensity of the secondary electrons was utilized in the present embodiment; however, it would also be possible to utilize the oscillation of the absorption current indicated in the fourth embodiment. In this case, the secondary electron detector 14 becomes unnecessary, so that the construction of the apparatus as a whole is further simplified.

[0029] Furthermore, in the above description, the irradiation of the sample with an electron beam was performed over the entire surface of the sample; however, [the present invention] can be similarly worked in cases where the electron beam is constricted, so that the film thickness in a specified region of the sample is measured.

[0030]

[Effect of the Invention] In the present invention, the film thickness of a sample during film formation by vacuum deposition can be measured and controlled with a precision corresponding to the thickness of a monatomic layer or better by irradiating the sample with the angle of incidence of the primary electrons set at an angle that is greater than the total-reflection angle. Accordingly, the following effects are obtained: namely, the construction of the overall apparatus can be simplified, handling of the apparatus is facilitated, and the cost of the overall apparatus can be reduced. In the present invention, furthermore, a low-energy electron gun whose structure is relatively simple compared to that of a conventional device using reflective high-speed electron diffraction is used. Accordingly, the following merit is also obtained: namely, the overall apparatus comprises an inexpensive system compared to cases where a special high-voltage power supply, electron gun and electron-optical system are used. The applicability of these effects in scholarly fields, and the industrial value of these effects, are extremely high.

[Brief Description of the Drawings]

[Figure 1] Figure 1 is a diagram which shows the basic construction of the present invention.

[Figure 2] Figure 2 is a diagram of the oscillation of the secondary electron intensity that is obtained in the present invention.

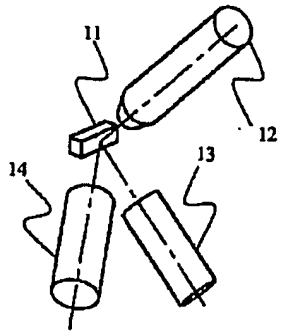
[Figure 3] Figure 3 is a diagram showing the combination of an energy analyzer with the present invention.

[Figure 4] Figure 4 is a diagram showing the construction used in a case where the oscillation of the absorption current is used in the present invention.

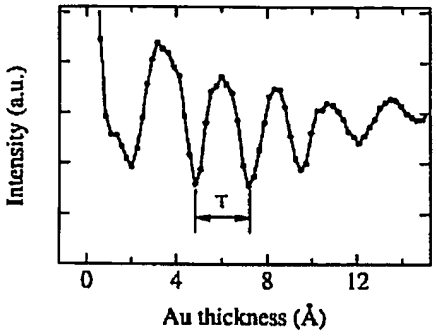
[Figure 5] Figure 5 is a diagram showing the automatic control of vapor deposition by the present invention and a computer.

[Explanation of Symbols]

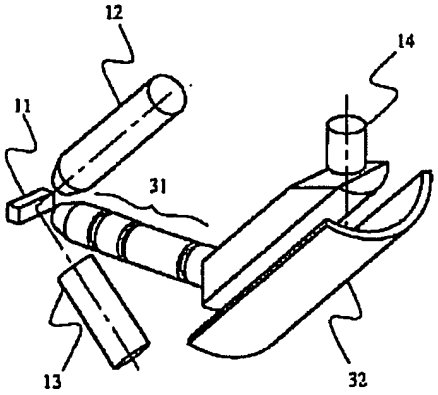
11... Substrate; 12... Electron gun; 13... Vapor deposition apparatus; 14... Secondary electron detector; 31... Electron lens; 32... Energy analyzer; 51... Computer.



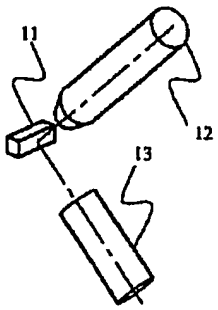
[Figure 1]



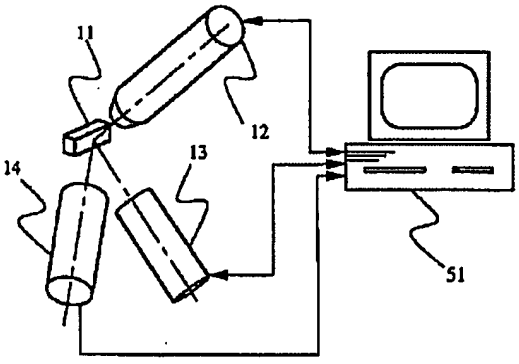
[Figure 2]



[Figure 3]



[Figure 4]



[Figure 5]